B. Implementing Agreement for a Programme of Research and Development on Advanced Materials for Transportation Applications

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Objectives

- Facilitate the integration of new technologies into the transportation sector by implementing research that validates the applicability of this technology to improve material properties while maintaining acceptable life-cycle costs.
- Promote commercialization of new materials technologies by developing standard testing and characterization methods in conjunction with national and international standards communities.

Approach

- Define and implement research under the International Energy Agency (IEA) Implementing Agreement (IA) entitled Implementing Agreement For A Programme Of Research And Development On Advanced Materials For Transportation Applications (IA-AMT).
- Conduct major research themes as annexes under the current IA:
  - Annex II: Co-Operative Program on Ceramics for Advanced Engines and Other Conservation Applications
  - Annex III: Co-operative Program on Contact Reliability of Advanced Engine Materials
  - Annex IV: A Cooperative Program on Integrated Engineered Surface Technology

Accomplishments

- Completed final work on project Annex II, "Subtask 13: Burner Rig Round Robin." The final report can be found at http://ia-amt.ornl.gov/index.html.
- Completed a final report summarizing all activities conducted under Annex II. The final report can be found at http://ia-amt.ornl.gov/index.html.
- Completed preliminary assessment of the laser spallation test for the measurement of coating adherence. The technique was capable of generating controlled spallation of both metallic and ceramic coatings. The remaining challenge is to quantify stress levels associated with the spallation event.

Future Direction

- Develop a plan for the lightweighting materials annex and present it to the Executive Committee for approval.
- Expand the scope of the implementing agreement by adding an annex on characterization/qualification of ceramic coatings for wear, thermal, and environmental protection.

Introduction

The current mission of the IA-AMT is to investigate promising new technologies for evaluating and ultimately improving the performance of materials for transportation systems. The primary motivation for this activity is the fact that new material technologies are required to increase efficiency and reduce harmful emissions in these systems. Examples of these technologies include (1) lightweighting to improve fuel efficiency; (2) surface engineering to improve the resistance to wear and contact damage; (3) development of durable coating systems for thermal, wear, and environmental management; and (4) development of revolutionary materials (structural ceramics and ceramic matrix composites) for operation at much higher temperatures and pressures. As discussed in this report, the research activities within the IA-AMT focus specifically on (1) identifying promising new technologies for improving materials performance and (2) developing specialized characterization techniques for validating the applicability of this technology to improve material properties while maintaining acceptable life-cycle costs.

Approach

In performance improvement, the current emphasis is on integrated engineered surface technology (IEST) and lightweighting of materials. IEST encompasses the synthesis, processing, characterization, and application of technologies that enhance the functionality of surfaces in contact with the environment or with the surfaces of other solids. Activities on lightweighting of materials focus on aluminum, high-strength steels, magnesium, metal and polymer composites, titanium, intermetallic alloys and other advanced materials. Current topics under consideration include (1) data on production and resource availability; (2) life cycle data on environmental impacts associated with production, processing and use of lightweight materials; (3) recycling information, including regulatory frameworks; (4) data on crashworthiness, design, and testing methodologies, (5) data on base material costs; (6) data on energy impacts of lightweight materials; and (7) shared information on research programs on lightweight materials.

In terms of performance evaluation, the primary focus is on techniques for (1) assessing environmental degradation of structural (non-oxide) ceramics; (2) evaluating time-dependent degradation of mechanical performance of structural ceramics; (3) quantifying key properties of coatings for wear, thermal, and environmental protection of current transportation materials; and (4) developing techniques for the measurement of key properties (topography, chemistry, subsurface damage) of engineered surfaces. Items 1 and 2 are motivated by the need to address key barriers to the use of this important class of material. For example, given the recent concern over environmental degradation of non-oxide ceramics in combustion environments, cost-effective techniques are required to simulate these effects as well as to assess the effectiveness of environmental barrier coatings. The IA-AMT is currently evaluating a variety of techniques ranging from complex high-pressure burner rig tests to a simple cost-effective steam injection system. Ceramic coatings hold considerable promise for (1) improving wear resistance, (2) providing thermal protection, and (3) reducing environmental degradation of critical metallic components used in internal combustion engines. Unfortunately techniques for assessing key properties particularly with respect the interface are unproven. Item 3 addresses this limitation. In a similar fashion, as surface modification technologies mature, proven characterization techniques will also be required to validate their performance (Item 4).

Results

A major result this year was the completion of Annex II. The Annex II subtasks primarily focused on addressing key technology barriers to the use of structural ceramics in both automotive and land-based gas turbines. The motivating factors for ceramic gas turbine development were the (1) desire to dramatically improve efficiency through higher turbine inlet temperatures, (2) potential for reducing the dependency on strategic metals, and (3) lowering of harmful emissions. Structural ceramics were considered for use as combustors, transition sections, hot gas ducts, turbine vanes and shrouds, turbine blades, and integrally bladed rotors (blisks). High-performance SiN and SiC ceramics were considered as leading candidates for use in these applications.[1–10] These materials offered the following advantages over their metallic counterparts: (1) low density, (2) refractoriness, (3) adequate short-term strength, and (4) good oxidation resistance at ambient pressures. The technical barriers to the implementation of ceramic technology in gas turbines included maturation of powder processing methodologies for component fabrication; establishment of proven methodologies to assess key mechanical properties (required for the development of design database); and optimization of the machining process used in the final stages of component manufacturing (related to cost optimization). Given these barriers, Annex II focused on (1) characterization of powder properties, (2) quantification of the green-state characteristics, (3) evaluation of mechanical performance (strength and thermal shock), (4) machining, and (5) evaluation of environmental degradation of non-oxide ceramics (see Table 1). An extensive set of results was generated from the work

Table 1. Summary of all subtasks implemented under Annex II

Subtask	Title (final report)	Started	Completed	Countries
1	Information Exchange	1985	2004	Germany, Japan, Sweden, United States
2	Characterization of Ceramic Powders: Data and Analyses	1985	Final Report—March 1990	Germany, Sweden, United States
3	Characterization of Sintered Silicon Nitride and Silicon Carbide Structural Ceramics	1985	Final Report—October 1989	Germany, Sweden, United States
4	(1) Fractography Analysis of Silicon Nitride and Silicon Carbide Structural Ceramics, (2) Statistical Analysis of Flexure Strength Data, and (3) Analysis of Error Sources in Four Point Flexure Strength Measurements of Structural Ceramics	1985	Final Reports—June 1989 (1 and 2) December 1989 (3)	Germany, Sweden, United States
5	Study of the Flexure and Tensile Strength of a United States Silicon Nitride	1990	Final Report—September 1993	Germany, Japan, Sweden, United States
6	Development and Testing of Procedures for Characterization of Ceramic Powders	1990	Final Report—September 1993	Germany, Japan, Sweden, United States
7	Effect of Machining Conditions on the Strength of Silicon Nitride	1993	Final Report—March 1998	Germany, Japan, Sweden, United States
8	Development and Testing of Primary and Secondary Properties of Ceramic Powders	1993	Final Report—December 1996	Belgium, Germany, Japan, Sweden, United States
9	Thermal Shock Testing of Advanced Ceramics	1996	Final Report—March 2000	Belgium, Germany, Japan, Sweden, United States
10	Assessment of Powder Characterization Methods for Advanced Ceramics	1996	Final Report—February 2000	Belgium, Germany, Japan, Sweden, United States
11	Techniques for the Measurement of Thermal and Mechanical Fatigue	2000	Final Report—March 2004	Germany, Japan, Sweden, United States
12	Characterization of Ceramic Powders and Green Bodies	2000	Final Report—September 2001	Belgium, Germany, Japan, Sweden, United States
13	Burner Rig Round Robin	2003	Final Report—March 2005	Germany, Japan, United States

conducted in Annex II. The major reports summarizing most of the subtasks can be downloaded from http://ia-amt.ornl.gov/index.html.

The results from Annex II were subsequently used in the establishment of standards [via American Society for Testing and Materials (ASTM), Japan Industrial Standards (JIS), Committee for European Normalization (CEN), International Organization for Standardization (ISO), and National Institute of Standards and Technology (NIST) guidelines (see the publications list). Table 2 summarizes some the existing standards that benefited from this work. In the case of room-temperature flexural strength, the standard, ASTM C1161, was revised to reflect lessons learned about fixturing and test specimen configurations. ISO 14704 evolved from several standards (ASTM C1161, CEN EN843-1 and JIS R1601) and lessons learned from the IA-AMT work. The high-temperature flexural strength standard, ASTM C1211, evolved about the time of the IEA round robin and included lessons learned from C1161 and the IEA work. ISO DIS 17565 (not yet a standard) has evolved from several standards (ASTM C1211, CEN prEN820-1, JIS R1604) and lessons learned from the IA-AMT subtask. The room-temperature tensile strength standard, ASTM C1273, was developed as the IA-AMT results were being reviewed and analyzed and included many lessons learned. ISO 15490 evolved from two standards (ASTM C1273 and JIS1606), and the ISO round robin results were instrumental in establishing test specimen configurations, gripping arrangements, allowable bending, and test rates. The thermal shock standard, ASTM C1525, was developed after the IA-AMT subtask on thermal shock was completed. Although this standard follows a more conventional approach to thermal shock by using water and standard MOR bars, insights garnered from the IA-AMT work are used in providing guidance to users in notes and discussions.

Thirteen subtasks were ultimately conducted under Annex II. Technical experts in the field defined the work scope for each subtask, with final approval given by the executive committee members during the annual meetings. Once the subtask was approved, each country was responsible for selecting technical task leaders; the executive committee appointed an overall subtask coordinator. Working group meetings were used to monitor the progress of the effort and provide a venue for technical data exchange by the task leaders. Once a subtask was completed, the coordinator was responsible for collecting and analyzing the data and preparing the final reports. Many of the reports are available on the IA-AMT web site: http://ia-amt.ornl.gov/index.html.

Table 2. Standards that have benefited from the IA subtasks

Property	JIS	ASTM	CEN	ISO
Flexural strength: RT	R1601-95	C1161-96	EN 843-1:95	14704:2000
Flexural strength: HT	R1604-95	C1211-98	prEN 820-1	DIS 17565
Statistical analysis	R1625-96	C1239-95	ENV 843-5:97	CD 20501
Fractography		C1322-96	prENV843-6	
Tensile strength	R1606-95	C1273-95		15490:2000
Sample preparation for the determination of particle size distribution of ceramic powders	R1619:95	C1282-94	EN 725-5:96	14703:2000
Surface area	R1626-96	C1274-95	EN 725-6:96	DIS 18757
Particle size distribution of powder by laser diffraction method	R1629:97			TC206 NP02
Thermal shock	R1615-93	C1100	prEN 820-3	
Flowability	R1639-4:99			TC206 WI93
Size distribution of granules	R1639-1:99			
Binder content of granules				
Drying loss of granules	R1639-3:99			
Bending fatigue: RT	R1621:95			TC206 PWI 07
Bending fatigue: HT	R16xx:01			

RT = room temperature; HT = high temperature.

As shown in Table 1, the 13 subtasks included an ongoing information exchange (subtask 1), which occurred in conjunction with the annual executive committee meeting, and specific research activities (subtasks 2–13), which consisted of task sharing collaborations between the participating national organizations. Most of these subtasks included extensive industry participation. Because the more recent subtasks involved fairly specialized hardware (thermal shock facilities for the centrally heated disk specimen—subtask 9, burner rig facilities—subtask 13), industrial participation was limited. Throughout Annex II, the industrial participants provided (1) expertise in the development of experimental research plans, (2) materials and facilities required for the implementation of those plans, and (3) personnel to actively participate in the research activities. In this approach, each country benefited from the experience and work of the other countries by gaining access to a larger quantity of data required to substantiate the standard methods under development. This approach also minimized the effort required to transition standards from the national to international level. As a result the structure of the IA led to considerable cost savings.

The motivation for subtask 13, the last task implemented under Annex II, was the need to develop a reliable method to assess the environmental attack on non-oxide ceramics arising from the global removal of the normally protective silica scale via reaction with water vapor present in the combustion gases. In stagnant environments, oxidation of both SiN and SiC is increased by (1) the replacement of oxygen by water vapor and (2) an increase in the pressure of the oxidant. In addition, the high velocities and presence of water in the environment can lead to the volatilization of the normally protective silica layer. The resulting recession of non-oxide ceramics such as SiC and SiN ultimately leads to excessive material loss, which increases the stress levels arising from both thermal and mechanical loading (Figure 1).

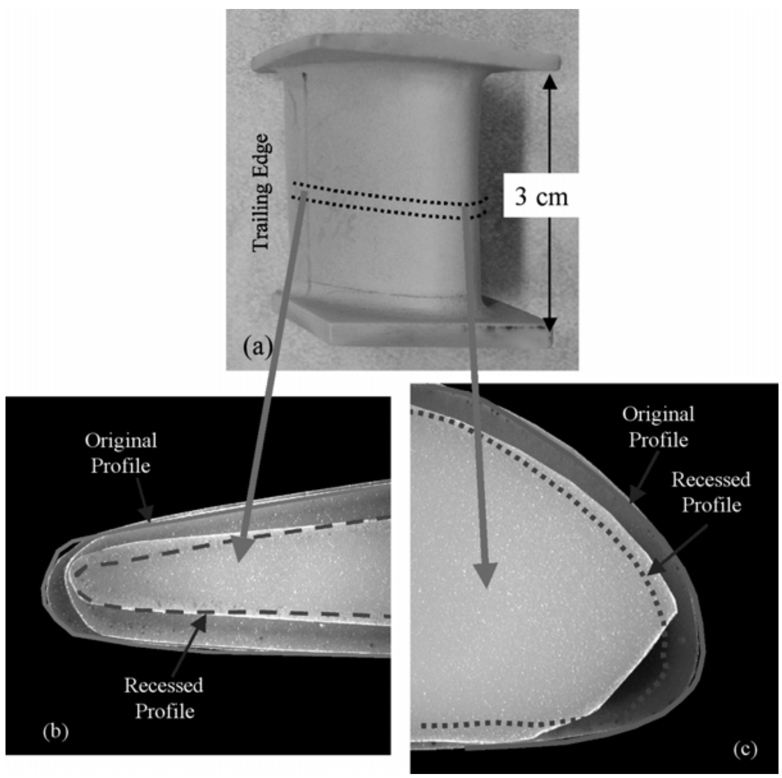

Figure 1. Photograph of uncoated AS800 prior to field testing (a). The dotted lines show the sections that were machined for subsequent polishing and evaluation by scanning electron microscopy. Typical sections for an as-received vane and a vane exposed for 815 h are compared in (b) trail edge and (c) leading edge. The dotted lines are predicted recession profiles.

Because of this recession problem, considerable emphasis has been placed on the development of environmental barrier coatings (EBCs) to act as diffusion barriers to water vapor. The effectiveness of EBC systems is defined by their (1) ability to isolate the non-oxide ceramic from the water vapor and (2) phase stability in the gas turbine environment. Both aspects can be evaluated from burner rig studies. Unfortunately, the results obtained from a given burner rig system depend upon a number of factors, including specimen geometries, gas flow/specimen configuration, temperature uniformity, and burner stability. The goal of this subtask was to implement a round robin in which a selected SiN material was evaluated in burner rig facilities located in Japan and the United States.

In subtask 13, three techniques were used to evaluate the recession behavior of the SN282 material. The first method consisted of exposing rectangular specimens for 400, 600, and 1000 hours in a high-pressure, high-velocity burner rig located at General Electric Global Research Center. This rig, which was used extensively used in the Continuous Fiber Ceramic Composite program,[11] runs largely unattended with extensive computer control, data acquisition, and safety monitoring. The second technique considered involved the development of a system for injecting steam onto a small region of the SiN specimen using an alumina or SiC tube. As shown in Figure 2, this method was quite effective at inducing localized recession in the injection area. The final technique involved exposing prismatic bars in an atmospheric burner rig. A detailed comparison of the resulting recession data[12] indicated that all three techniques gave comparable values of the recession rates (Figure 3).

Conclusions

The IA-AMT has made significant progress in expanding its scope, as evidenced by the addition of two new annexes, IV and V. Both annexes are expected to generate new members. Given this progress, a formal request has made to the IEA to extend the IA-AMT for a period of 3–5 years.

Presentations and Publications

M. K. Ferber, *Burner Rig Round Robin—Subtask 13: Final Report*, March 2005. Available at http://ia-amt.ornl.gov/index.html.

M. K. Ferber, *Annex II: Co-Operative Program on Ceramics for Advanced Engines and Other Conservation Applications—Final Report*, September 2005, available at http://ia-amt.ornl.gov/index.html.

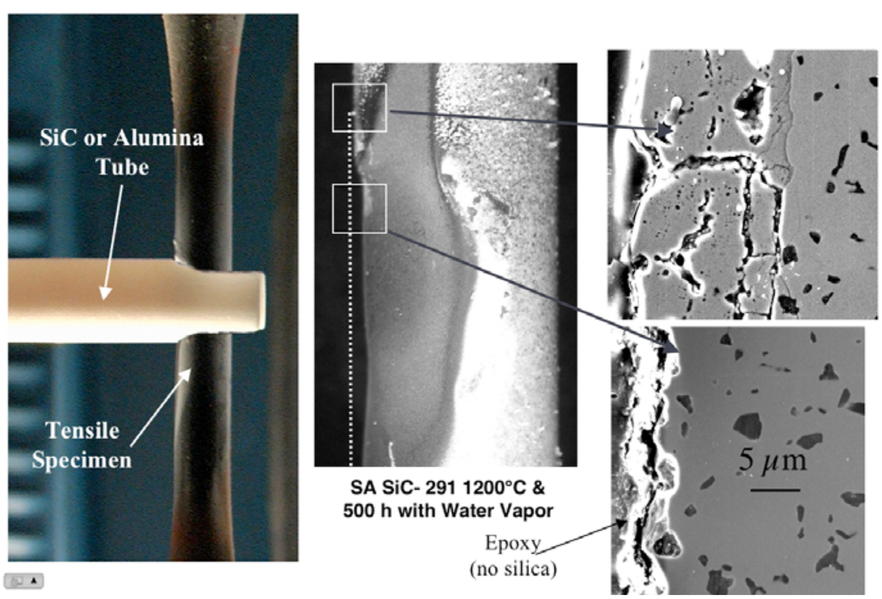

Figure 2. Sintered alpha SiC tensile specimen after 500 h exposure to a saturated water vapor environment at 1200°C. The white dotted line on the right shows the position of the original surface prior to testing.

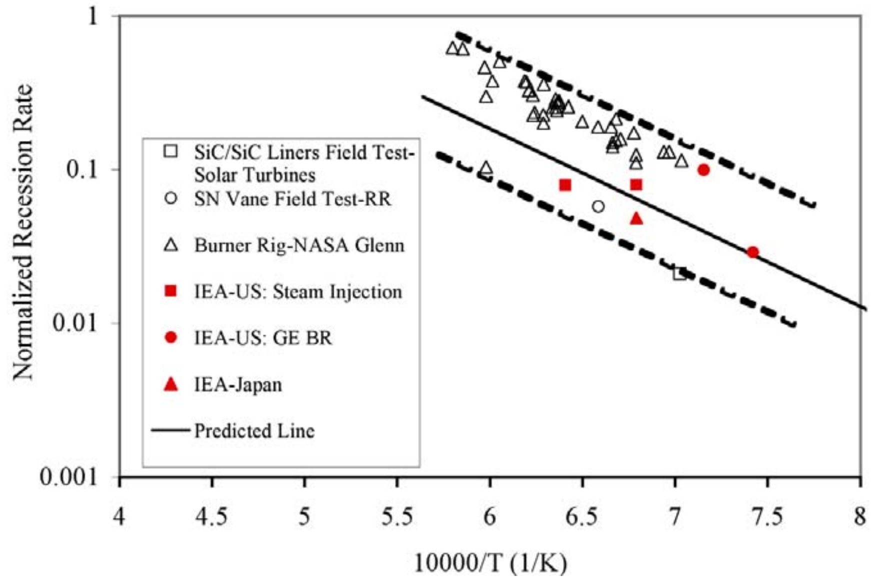

Figure 3. Comparison of normalized recession rate versus 1/T data obtained in present study with similar data reported in the literature.

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